UJEAS

UNIZIK JOURNAL OF ENGINEERING
AND
APPLIED SCIENCES

UNIZIK Journal of Engineering and Applied Sciences (2023), 69 - 77
Journal homepage: https://journals.unizik.edu.ng/index.php/ujeas

Effects of rice straw fibre and walnut shell ash particulates on the mechanical behavior of epoxy composite

Chilee Ekwedigwe[1], *Chidume Nwambu[1] and Eugene Nnuka[1]

[1]Department of Metallurgical and Materials Engineering, Faculty of Engineering, Nnamdi Azikiwe University, Awka, Anambra State, Nigeria

*Corresponding Author's E-mail: cn,nwambu@unizik.edu.ng

Abstract
This work studied the effects of rice straw fibre and walnut shell ash particulates on the tensile and impact behaviours of epoxy composites. Conventional hand-lay-up techniques in five different weight proportions (i.e 2 wt.%, 4wt.%, 6wt.%, 8wt.% and 10wt.%) were used to reinforce the epoxy composites. The rice straw fibres were used to reinforce the epoxy composite separately, while rice straw fibres combined with walnut shell ash particulates at an equal ratio were also used to reinforce the composites. The composites were tested in tension (according to ASTM D3039) and impact (according to ASTM D256). Both stiffness and strength were positively affected by reinforcement of the epoxy composites with rice straw fibre and walnut shell ash particulate. Amongst the different weight proportions, 6wt% of hybrid composite (rice straw fibre and walnut shell ash particulate) gave the highest tensile strength and damage tolerance. These developed epoxy biocomposites with high tensile strength and toughness, made with agricultural waste reinforcers, could be a sustainable, affordable, and environmentally friendly alternative material for automotive, structural and defense applications.

Keywords: Composite, epoxy resin, rice straw, walnut shell ash particulate, tensile and impact strength.

1. Introduction

Wind energy generation has a low impact on the environment and is a good option for locations where traditional energy distribution networks do not reach, but the overall performance of a wind turbine system depends on the quality of the blades, and the failure of the blades ultimately results to system failure, Abdul-Nasir *et al.,* (2014), Debnath *et al.,* (2013). In order for blades to withstand the static and dynamic loads they are subjected to, superior mechanical performance is required. In light of these requirements, long fibre reinforced polymer composites proffer an ideal solution since they have high specific strength, high stiffness, corrosion resistance, and low density, Rabbani *et al.*, (2022), Bisht *et al.*, (2020). Although glass and carbon fibre are the primary reinforcement materials for wind turbine blades, natural fibre can also serve as a blade material for micro wind turbines, Kuram (2020) Yadav *et al.*, (2021). As a result of their inherent superior properties, synthetic fibre-reinforced polymer composites have been incorporated into a wide range of applications, including the construction, aerospace, and automobile industries, Nwambu *et al.*, (2022), Ibrahim *et al*., (2010). However, these composites are non-biodegradable, which poses a significant threat to the ecological system, Braga *et al*., (2015). In addition, synthetic fibres, such as glass and carbon, pose several health risks. The need for lightweight materials coupled with strict environmental rules and regulations have led to the development of natural fibre reinforced biocomposite materials, Arifuzzaman-Khan *et al.,* (2016). Because natural fibre polymer composites are lightweight, they are widely accepted in the automobile industry due to the direct effect on the fuel efficiency of vehicles, Chandrasekaran *et al*., (2014).

Several researchers have investigated the effect of natural fibres on the mechanical properties of the epoxy composite. For example, Rabbani *et al.,* (2022) characterised the mechanical properties of the biocomposite by using the polypropylene random copolymer (PPRC), and biowaste rice husk (BRH) as the primary raw materials. They found that the modulus and hardness of the biocomposite improved by 44.8% and 54.8% due to the neat PPRC, respectively. Also, Kuram (2020), studied the effect of natural filler on the morphological, rheological and mechanical properties of acrylonitrile–butadiene–styrene (ABS) terpolymer. The powdered hazelnut and walnut shells were employed as natural filler with ABS to develop hybrid polymer composites. Their findings revealed that the highest strengths and flexural modulus were achieved with 5wt% of hazelnut and 15wt% of walnut shell four among all hybrid composites. Bisht *et al*., (2020) attempt to understand the applicability of rice husk as a fibre with various polymers composites. They highlighted various modification techniques that can enhance the mechanical properties of biocomposite reinforced with rice husks by altering their chemical and physical properties. Okafor *et al.* (2022) investigated the tensile behaviour of *Dioscorea alata* stem fibre/epoxy composites (DASFEC) after extraction and treated with two percent concentration of Sodium hydroxide (NaOH) for four hours. They reported that the longitudinal tensile strength of 94.2MPa indicates an improvement in the tensile performance of the developed composite. Yadav et al. (2021) proposed that utilising various weight percentages of rice husk ash and eggshell as reinforcement material with aluminum can improve the mechanical properties of composites. Alshahrani and Prakash (2022) investigated the role of adding bio toughener and reinforcements in making biocomposite for sustainable engineering applications. Areca fibre, rice husk ash (RHA) biochar and cardanol oil were prepared by low-temperature pyrolysis and Soxhlet extraction. According to the mechanical test, adding cardinal oil to the resin improved the toughness (7.2J), tensile strength (208 MPa), and flexural strength (236 MPa). Analyses of bio-fibre comminution energy and particle size classification were conducted as the first step toward converting cellulose-based agricultural residues into environment-friendly composite products, Okafor *et al*., (2022).

Recent global directives such as 2009/33/EC and 2008/98/EC, Obande *et al*., (2019), stipulate strict weight-reduction targets for transport and cross-sectoral reduction of landfill-bound waste streams, natural fibres-based reinforced composites present an outstanding alternative to synthetic materials. These natural fibres are rice straw and walnut shell ash. Rice straw, as a lignocellulosic biomass, comprises three components: lignin, cellulose, and hemicelluloses. These could be fractionated through pretreatment, Saidah *et al*., (2017). Walnut shell is a waste generated in the walnut (*tetracarpidium conophorum*) harvest, containing natural antioxidant compounds such as flavonoids, Akbari *et al*., (2012). Much environmental pollution is involved in disposing of these natural fibres, kuram (2020). Thus, it is necessary to find new ways to use these natural fibres which will prevent environmental concerns and provide a second income for farmers, Okafor *et al.*, (2022). Therefore, this paper will focus on reinforcing the epoxy composites with rice straw fibre and walnut shell ash particulate to improve their mechanical properties.

2.0 Material and methods

2.1 Materials

The materials used in the experiment include epoxy (LY556) bisphenol-A-diglycidyl- ether, hardener (HY-951), sodium hydroxide solution and acetic acid solution sourced from Lagos. Rice straw fibres and walnut shells were obtained from Abakiliki, Ebonyi state. At the same time, petroleum jelly and distilled water were from the Chemistry Department, University of Nigeria, Nsukka. All the equipment used in the experiment are as follows; vibratory sieve shaker, electric oven, weighing balance, universal material testing machine Cat Nr. model:261 and SEM machine model: JEOL JSM840.

2.2 Chemical treatment of bio fibres

Rice straw fibres (RSf) were washed with regular water to remove dirt and air-dried at 25-30°C for 24 hrs and were treated with NaOH to improve the quality. Likewise, the walnut shell (WSAp) was washed, dried, and placed inside a perforated cylinder pan for combustion into ash. The obtained ash was then placed inside an alumina crucible, put inside a muffle furnace, heated to a temperature of 900°C and held for 5hrs to reduce the carbonaceous matter and increase the percentage of active silica content. The X-ray diffraction patterns of the WSAp and RSf was determined by Siemens D-500 diffractometer using Co-K_C radiation ($K_C = 1.79026$ A). The microscopic study of WSAp and RSf was determined by JEOL JSM840A scanning electron microscope (SEM) complemented by EDS. Particle size and morphology of produced WSAp were examined by TEM (Jeol, JSM2010), using a 200 keV electron beam on the sample mounted on a carbon-coated copper grid. The WSAp was a suspension taken using a dropper, spread on the carbon coated copper grid, and allowed to dry at room temperature. The copper grid was introduced into the instrument, and the sample chamber was evacuated. The sample was scanned along the path of the electron beam, and a photograph of the sample was taken. The elemental composition of the WSAp and RSf were determined by X-Ray Fluorescence (XRF) analysis. The samples were formed into pellets in a pelletiser with the hydraulic press (Carver Inc). The pellets were sealed at the chamber of the XRF (Amptek Inc) and allowed to run for 1000s at a voltage of 20 kV, and a current of 40μA.

2.3 Manufacturing procedure

Rice straw fibre (RSf) reinforced epoxy resin composites were fabricated by conventional hand-lay-up techniques in five different weight proportions (i.e 2 wt.%, 4wt.%, 6wt.%, 8wt.% and 10wt.%), while the hybrid composites were measured at the equal ratio of rice straw fibre (RSf) to walnut shell ash particulate (WSAp) and produced in five different weight proportions (i.e 2 wt.%, 4wt.%, 6wt.%, 8wt.% and 10wt.%). The WSAp was thoroughly dispersed in the matrix material using Digital Sonicator (Q-700-200, Qsonica, Newton, USA)) for homogeneity concerns. The low temperature curing epoxy resin and corresponding hardener were blended in a ratio of 10: 1 by weight as recommended by the supplier and available literature, Debnath *et al.*, (2013). The mixing was done meticulously before the rice straw fibre mats were reinforced in the matrix body. Fibre mats were cut in the dimension of 400mm × 400mm, and the wooden mould of 410 ×410×40 mm^3 was used to fabricate the composites. The composite slabs were fabricated by a modified hand lay-up method followed by a light compression moulding technique. After curing, easy release of composite from mould was assured by applying releasing agent (Silicon spray). Care was taken for WSAp powder-filled epoxy composites to ensure a uniform sample since solid particles tend to clump and tangle together when mixed. The curing of composites took place under a load of 25 kg for 24 hours before removal from the mould. Then, the composite slabs were post-cured in the air for another 24 hours after removal from the mould. For testing, specimens of the size specified by respective standards were cut from the prepared composite slabs using a diamond cutter. Homogeneity of the composite were maintained with the utmost care during fabrication.

2.4 Mechanical characterisation

Impact tests of the epoxy composites was conducted on a pendulum type impact testing machine specified by ASTM D256. The standard specimen size for ASTM D256 (63.5mm × 12.7mm × (2.5 – 5) mm with a 'V' notch at the center of length was used. In this test method, the notch produces a stress concentration that increases the probability of a brittle fracture rather than ductile. The specimen was held as a vertical cantilever beam and broken by a single pendulum swing. The initial contact line was at a fixed distance from the specimen clamp and the centerline of the notch, and on the same face as the notch. Likewise, the tensile test of reinforced polymer composite is generally performed on flat specimens. The dimension of the specimen is 250 mm ×25 mm × (4) mm according to the standard test method for tensile properties of fibre reinforced polymer composites ASTM D3039. During the test, a uniaxial load was applied through both the ends of the specimen at a crosshead speed of 10mm/min. The tensile test was performed in the Testometric testing machine, and the results were analysed to calculate the tensile strength of the composite samples.

3.0 Results and Discussions

3.1 Impact energy and percentage elongation of the composite

Figures 1a&b displayed the impact energy and percentage elongation of the composite samples. The impact energy and percentage elongation of the epoxy/RSf composites increase with increases in the wt%RSf from 0 to 8wt% ratio before decrease. Also, the impact energy and percentage elongation of the hybrid composite (epoxy/RSf +WSAp) increased to a maximum at 6wt% ratio of RSf+WSAp before decrease. According to the results, RSf (rice straw fibres) can provide no additional impact resistance to the epoxy polymer after 8wt% of addition. Likewise, the combination of walnut shell ash particulates and rice straw fibres fails to provide any additional impact resistance to the epoxy composites after 6wt% of addition. At this stage, the composites are becoming more brittle than at the early stage. The impact strength and percentage elongation of the composites are attributed to the strong and tough nature of the RSf, which helped the composites absorb more energy before failures. It was observed that hybrid composites (RSf + WSAp) have superior impact resistance compared to only rice straw fibre (RSf) composites. The highest values, 7.65 and 8.87 J/mm^{2,} of the improved impact energy of the hybrid composites (RSf + WSAp), are within the recommended standard for wind turbine blades (Debnath et al., 2013).

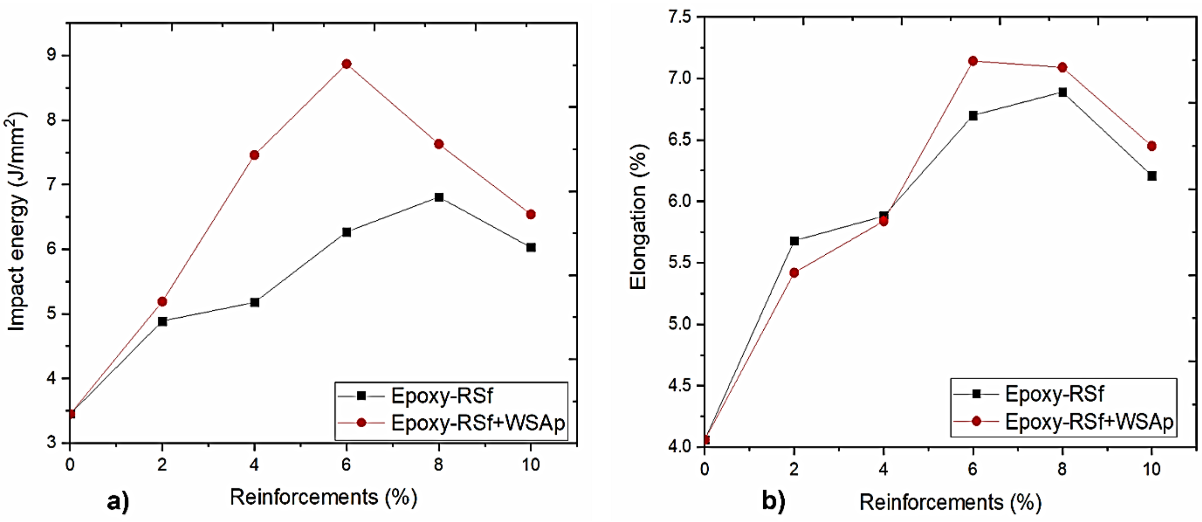

Figure 1: Effect of reinforcements on the (a) impact energy and (b) percentage elongation of the composite.

3.2 Tensile properties of the composite

The results of the tensile strength and stiffness of the composites are shown in Figures 2a&b. The hybrid composites (epoxy/RSf+ WSAp) have higher tensile strength and stiffness values compared to composites with only rice straw fibres (epoxy/RSf). It was seen clearly that the tensile behaviour and toughness increased with an increase in the percentage of reinforcement. The increment in the toughness of the composites could be attributed to the high stiffness of the rice straw fibres (RSf), which agrees with a similar result obtained from the work of Debnath *et al.*, (2013) on the reinforcement of epoxy polymer. The tensile strength of the composite samples with rice straw fibres (epoxy-RSf) increased as the weight percentage of rice straw fibre (RSf) increased from 2wt% to 8wt%. While for hybrid composites, epoxy-RSf+ WSAp, the tensile strength increased to its maximum at a 6wt% ratio before the decrease in the tensile strength. The increase in the tensile strength could be because of good interfacial bonding between the polymer and reinforcement. For example, a tensile strength of 91.38 MPa and 89.17 MPa and percentage improvement of 165.33% and 158.94% were obtained at 8wt% of RSf and 6wt% of RSf +WSAp. The tensile strength obtained at 8wt% of RSf and 6wt% of RSf +WSAp were within the recommended standard to produce wind turbine blades, Arifuzzaman-Khan *et al.*, (2016).

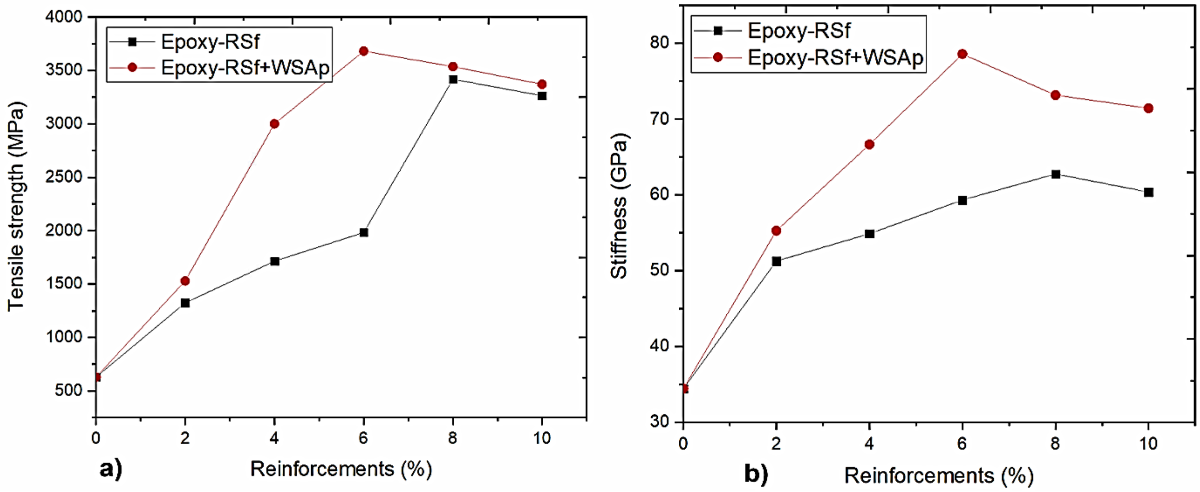

Figure 2: Effect of the reinforcements on the (a) tensile strength and (b) stiffness of the composites.

3.3 Fracture surface of the tensile sample

Figures 3-5 displayed the tensile fracture surface and showed a significant difference in fracture morphology of the samples under investigation. The fracture surface of the 100%epoxy with reinforcement showed a coarse and zig-zag surface (Figure 3). This was due to the brittle nature of the surface.

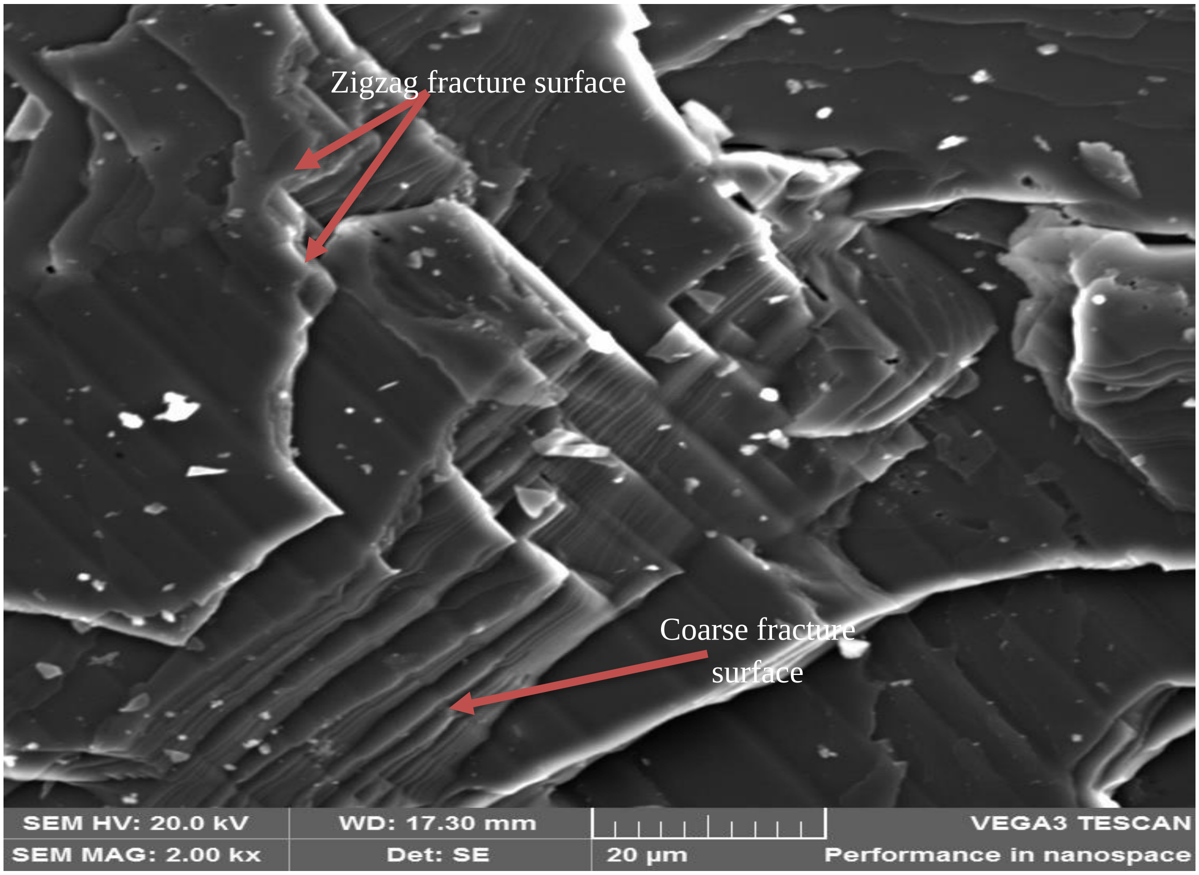

Figure 3: SEM tensile fracture surface of the composite sample with 100%epoxy.

The tensile fracture surface of the composites showed that the reinforcement was covered with epoxy. This could be attributed to good interfacial bonding between the reinforcement and the epoxy. This was the major reason why the tensile strength obtained in the reinforcement composites was higher than that of the pure epoxy composited under investigation. A similar observation of improvement in tensile strength of epoxy after reinforcement was reported by Nwambu *et al.*, (2022). However, when Figure 3 was compared to Figure 5, it became clear that there were some particle detachments from the epoxy-RSf+WSAp composites. These particle detachments were the reason behind the optimum tensile strength obtained in 6wt% of epoxy-RSf+WSAp.

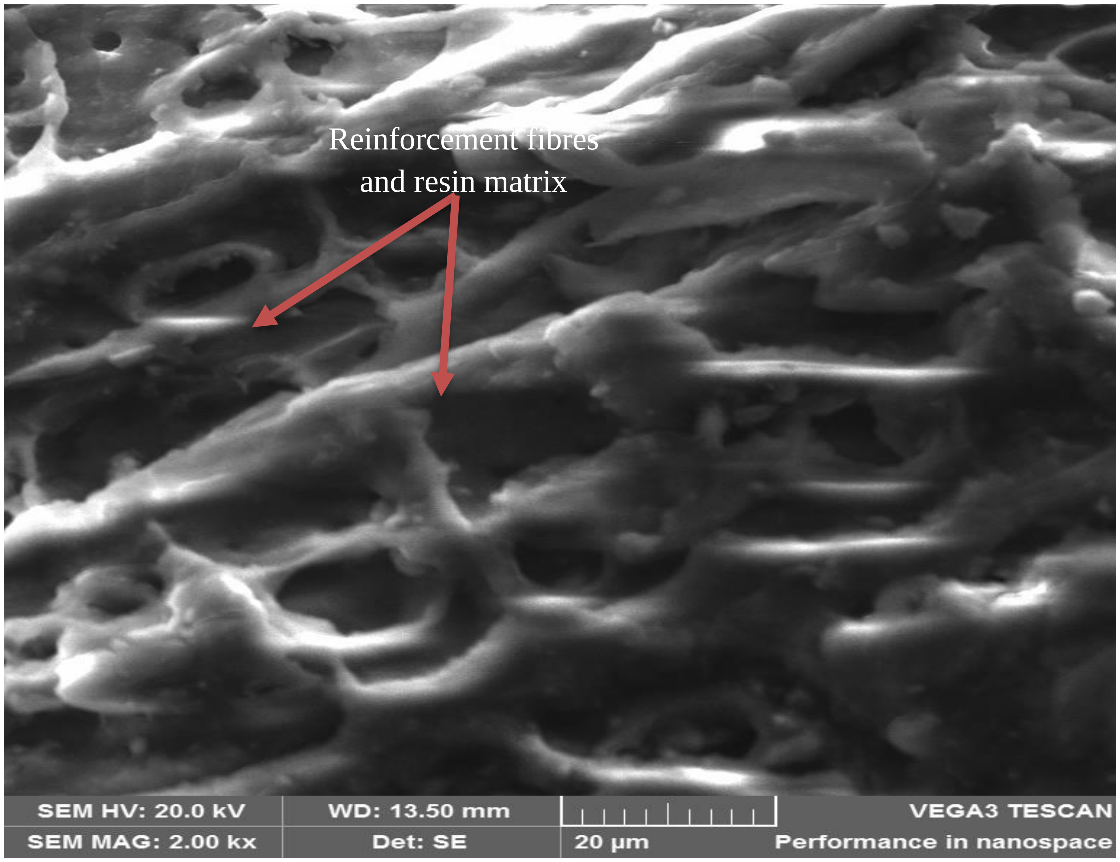

Figure 4: SEM tensile fracture surface of the composite sample with epoxy-8wt%RSf.

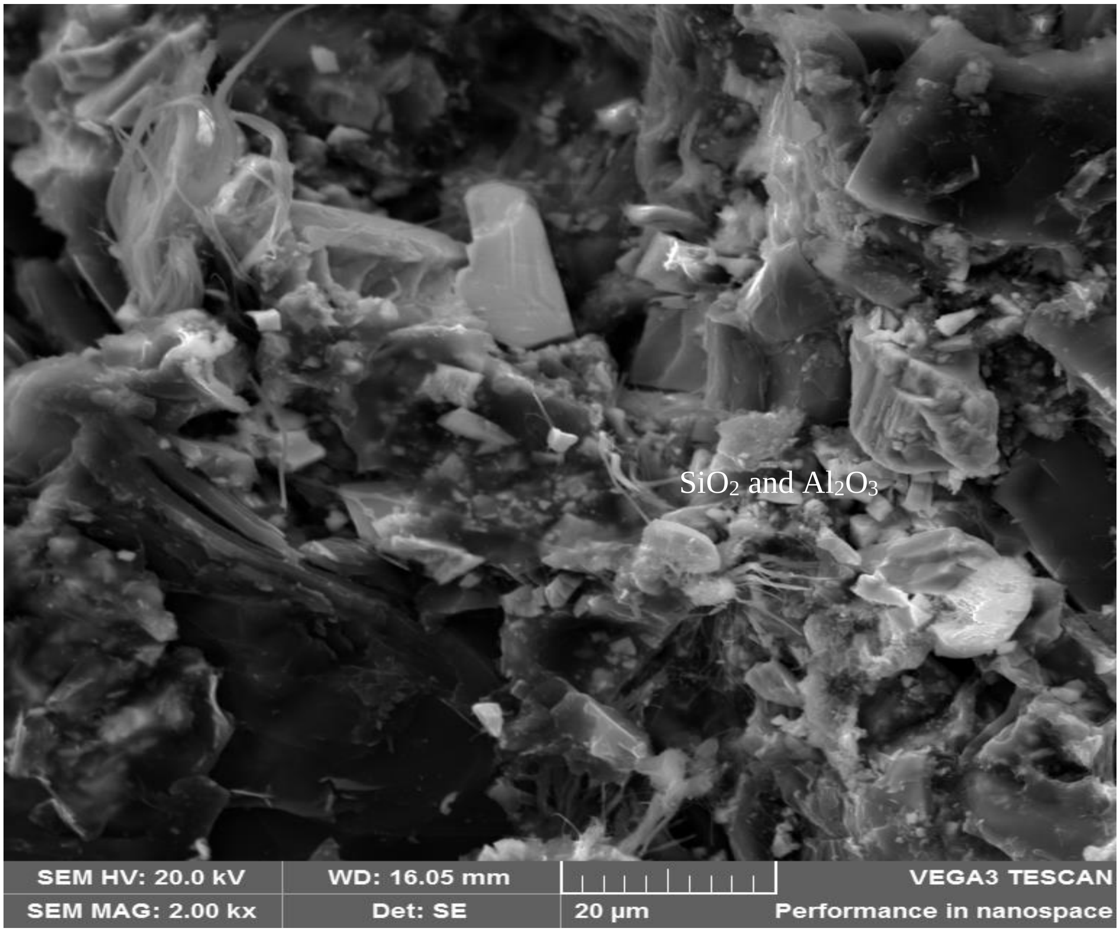

Figure 5: SEM tensile fracture surface of the composite sample with 6wt% of epoxy-RSf+WSAp.

The findings from this research might prove helpful in improving the performance of tidal turbine blades and automotive equipment in the future by selecting the appropriate material during production to optimise those systems' performance. In order to gain a complete understanding of the relationship between the microstructure and the mechanical behavior of particulate reinforced composites, the tensile and impact strength of the epoxy composite were investigated. Based on the analysis of the bio-fibres as reinforcement materials on the epoxy composite for improvement of mechanical properties, bio-fibres could be selected for use in eco-friendly composite construction. The findings of this research align with the reports of recent studies, Saidah *et al*., (2017), Okafor *et al.*, (2022), Nwambu *et al*., (2022) on the effects of bio-fibres on the mechanical behaviors of epoxy composite structures.

4.0. Conclusion

It was established that the reinforcements (rice straw fibre and walnut shell ash particulate) improved the mechanical properties of the composite. The improved tensile strength obtained at 8wt% of RSf and 6wt% of RSf+WSAp is within the recommended standard to produce wind turbine blades. Improvement of impact energy and %elongation of 83.77 and 75.07 % were obtained at 8wt% of epoxy-RSf and 6wt% of epoxy-RSf+WSAp composites. The hybrid composite samples can absorb and dissipate energy better at 8wt% of epoxy-RSf and 6wt% of epoxy-RSf+WSAp. These developed epoxy biocomposites with high tensile strength and toughness, made with agricultural waste reinforcers, could be a sustainable, affordable, and environmentally friendly alternative material for automotive, structural and defense applications.

References

Abas F.O., R.O. Abas, S.I. Ibrahim, A 2010. Comparison Study of Different Ceramic Filler on Mechanical and Thermal Properties of Glass, Carbon, Kevlar / Polyester Composites, Engineering & Technology Journal, 28 2469-2479.

Abdul-Nasir, A. A., Azmi, A. I. and Khalil, A. N. M. 2014. Investigating the effect of machining parameters on mechanical performance of Flax Natural Fibre Composites with Circular Holes; Conference: Advances in Material Processing Technologies.

Akbari, V., Jamei R., Heidari, R. and Esfahlan, A. J. 2012. Antiradical activity of different parts of Walnut (Juglansregia L.) fruit as a function of genotype; *Food Chemistry*; Vol. 135 No. 4 pp. 2404-2410.

Alshahrani, H. and Prakash, A. 2022. Mechanical, wear, and fatigue behaviour of alkali-silane-treated areca fiber, RHA biochar, and cardanol oil-toughened epoxy biocomposite; *Biomass Conversion and Biorefinery*.

Andi Saidah , Yudinata , Sri Endah Susilowat, Saidah, A. and Susilowati, S. E. 2017. Design of composite material of rice straw fiber reinforced epoxy for automotive bumper, 2017 International Conference on Computing, Engineering, and Design (ICCED). Pp. 1-4.

Arifuzzaman-Khan, G. M., Terano, M., Gafur, M. A. and Shamsul-Alam, M. 2016. Studies on the mechanical properties of woven jute fabric reinforced poly (L-lactic acid) composites; Journal of King Saud University – Engineering Sciences 28, 69–74.

Braga, R. A. and Magalhaes Jr P. A. 2015. Analysis of the mechanical and thermal properties of jute and glass fiber as reinforcement epoxy hybrid composites; Materials Science and Engineering, Vol. 56 pp. 269–273.

Chandrasekaran, S., Sato, N., Tolle, F., Mulhaupt, R., Fiedler, B. and Schulte, K. 2014. Fracture toughness and failure mechanism of graphene-based epoxy Composites; Composites Science and Technology, Vol. 97 pp. 90–99.

Debnath K., Singh, I., Dvivedi, A. and Kumar. P. 2013. Natural fibre reinforced-polymer composites for wind turbine blades: challenges and opportunities; Recent Advances in Composite Materials for Wind Turbines Blades, pp. 25-38.

Ibrahim, I. S., Abas, R. O. and Abas F.O. 2010. A Comparison study of different ceramic filler on mechanical and thermal properties of glass, carbon, kevlar / polyester composites; Engineering & Technology Journal, Vol. 28 No. 12 pp. 2469-2479.

Kuram, E. 2020. Rheological, mechanical and morphological properties of hybrid hazelnut (corylus avellana L.)/walnut (Juglans regia L.) shell four-filled acrylonitrile butadiene styrene composite; Journal of Material Cycles and Waste Management, Vol. 22 pp. 2107–2117.

Neeraj Bisht*, Prakash Chandra Gope, and Nisha Rani, Bisht, N., Gope, P. C. and Rani, N. 2020. Rice husk as a fibre in composites: A review; Journal of the Mechanical Behavior of Materials, Vol. 29: No. 1 pp.147–162.

Nwambu, C. N., Robert, C. and Alam, P. 2022. The tensile behaviour of unaged and hygrothermally aged asymmetric helicoidally stacked CFRP composites; *Journal of Composites Science*, Vol. 6 No. 137.

Nwambu, C. N., Robert, C. and Alam, P. 2022. Viscoelastic properties of bioinspired asymmetric helicoidal CFRP composites, MRS Advances, pp. 1-6.

Obande, W., Ray, D. and Bradaigh, C. M. O. 2019. Viscoelastic and drop-weight impact properties of an acrylic-matrix composite and a conventional thermoset composite – A composite study. *Matter Letters*, Vol. 238 pp. 38-41.

Okafor, C. E., Kebodi, L. C., Kandasamy, J. May, M. and Ekengwu, I. E. 2022. Properties and performance index of natural fiber reinforced cross-ply composites made from dioscorea alata stem fibers, *Composites Part C*, Vol. 7 No. 100213.

Okafor, C. E., Kebodi, L. C., Kandasamy, J., May, M. and Ekengwu, I. E. 2022. Properties and performance index of natural fiber reinforced cross-ply composites made from dioscorea alata stem fibers; *Composites Part C*, Vol. 7 pp. 100213.

Okafor, C. E., Onovo, A. C. and Ihueze, C. C. 2020. Predictive energy requirement models in bio-fibre comminution process; Applied Sciences, Vol. 2 pp. 2047.

Okafor, C. E., Onovo, A. C. Ani, O. I., Obele, C. M., Dziki, D., Ihueze, C. C. and Okonkwo, U. C. 2022. Mathematical study of bio-fibre comminution process as first step towards valorisation of post-harvest waste materials; *Cleaner Materials*, Vol. 4 No. 100067.

Rabbani, F. A., Yasin, S., Iqbal, T. and Farooq, U. 2022. Experimental study of mechanical properties of polypropylene random copolymer and rice-husk-based biocomposite by using nanoindentation; Materials, Vol. 15 No. 5 pp. 1956.

Rajat Yadav, Vijay Kumar Dwived, Shashi Prakash Dwivedi, Yadav, R., Dwived, V. K. and Dwivedi, S. P. 2021. Eggshell and rice husk ash utilisation as reinforcement in development of composite Material: A review; Materials: Today Proceedings, Vol. 43 No. 1 pp. 426-433